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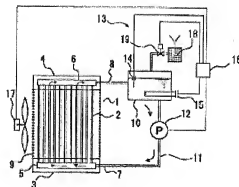
## (54) Name of Invention

High polymer film humidifier using  
hygroscopic hollow fiber

## (57) Summary

[Problem]

To provide hollow fibers having a function whereby moisture is adsorbed while any gas is passing through the hollow portion thereof, as well as a convenient gas humidifier using said hollow fibers.



[Means of resolution]

[Said] hygroscopic hollow fibers are hollow fibers having as base material an aromatic polyether sulfone polymer, or aromatic polyether amide polymer, or aromatic polyether amide alkyl sulfone polymer, and a solution of said polymer is ejected from an outer annular nozzle and caused to congeal by wet spinning wherein an inner annular nozzle ejects a core liquid of one or more of the following: water, or a copolymer suspension of tetrafluoroethylene and perfluorovinyl ether, or an aqueous polysulfonated styrene-Na solution. In [said] gas humidifier, a large multiplicity of hygroscopic hollow fibers 2 are

B12

arranged in line such that their middle parts remain independent while their ends are caused to be closely attached, such that [their] central cavity 2h fits against closed spaces 5 or 6 at either end, one such closed space 5 communicating to water intake port 7, and the other such closed space communicating with water outlet port 8.

### Scope of Claim for Patent

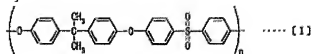
#### Claim 1

A high polymer film humidifier using hygroscopic hollow fiber, whereby hollow fibers having as base material an aromatic polyether sulfone polymer, or aromatic polyether amide polymer, or aromatic polyether amide alkyl sulfone polymer, and formed whereby a solution of said polymer is ejected from an outer annular nozzle and caused to congeal by wet spinning wherein an inner annular nozzle ejects one or more of the following: water, or a copolymer suspension of tetrafluoroethylene and perfluorovinyl ether, or an aqueous polysulfonated styrene-Na solution, are used in [said] gas humidifier, whereby a large multiplicity of hygroscopic hollow fibers are arranged in line such that their middle parts remain independent while their ends are caused to be closely attached, such that [their] central openings fit against closed spaces at either end, one such closed space communicating to a water intake port, and the other such closed space communicating with a water outlet port.

#### Claim 2

A high polymer film humidifier using hygroscopic hollow fiber according to Claim 1 whereby said aromatic polyether sulfone polymer is a polymer having repetitions of Chemical Formula I below:

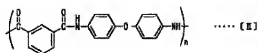
[Chemical Formula 1]



#### Claim 3

A high polymer film humidifier using hygroscopic hollow fiber according to Claim 1 whereby [said] aromatic polyether amide polymer has repetitions of Chemical Formula II below:

[Chemical Formula 2]



### Detailed Description of Invention:

0001

Field to Which the Invention Belongs

The present invention relates to a high polymer film humidifier used as a filter for a humidifier.

0002

#### Prior Art

Humidifier technology according to prior art was primarily by means of misting methods or ultrasound methods, or by means of electric heaters, requiring substantial energy and particularly with misting methods and ultrasound methods having problems in that impurities in water were dispersed at the same time.

0003

#### Problem to be Resolved by the Invention

Accordingly, the purpose of the present invention is to provide a hollow fiber with exceptional moisture adsorption properties, as well as a low-energy, clean (microbe-free) humidifier using such a hollow fiber.

0004

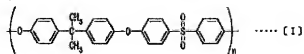
#### Means of Resolving the Problem

The present invention achieves the aforementioned purpose by means of a high polymer film humidifier using hygroscopic hollow fiber, whereby hollow fibers having as base material an aromatic polyether sulfone polymer, or aromatic polyether amide polymer, or aromatic polyether amide alkyl sulfone polymer, and formed whereby a solution of said polymer is ejected from an outer annular nozzle and caused to congeal by wet spinning wherein an inner annular nozzle ejects one or more of the following: water, or a copolymer suspension of tetrafluoroethylene and perfluorovinyl ether, or an aqueous polysulfonated styrene-Na solution, are used in [said] gas humidifier, whereby a large multiplicity of hygroscopic hollow fibers are arranged in line such that their middle parts remain independent while their ends are caused to be closely attached, and such that [their] central openings fit against closed spaces at either end, one such closed space communicating to a water intake port, and the other such closed space communicating with a water outlet port.

0005

The aforementioned aromatic polyether sulfone polymer preferably is a polymer having repetitions of Chemical Formula I below.

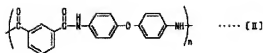
[Chemical Formula 3]



0006

Also, the aromatic polyether amide polymer preferably is a polymer having repetitions of Chemical Formula II below.

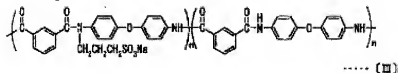
[Chemical Formula 4]



0007

Accordingly, the aromatic polyether amide alkyl sulfone polymer has repetitions of Chemical Formula III below.

[Chemical Formula 5]



0008

### Forms of Implementation of the Invention

Also, air humidifier 1 according to the present invention, formed in order to achieve the aforementioned purpose, as shown in Fig. 1 corresponding to a preferred embodiment, has a large multiplicity of aforementioned hygroscopic hollow fibers 2 are arranged in line such that their middle parts remain independent while their ends are caused to be closely attached, and such that [their] central openings fit against closed spaces 5 and 6 at either end having respectively input cap 3 and output cap 4, one such closed space 5 communicating to water intake port 7, and the other such closed space 6 communicating with water outlet port 8.

0009

In this air humidifier, water whereto pressure and heat are applied enters from water intake port 7 into closed space 5, then enters through the ends of multiple hollow fibers 2 into central cavity 2h (see Fig. 3), passes through, goes from the other end into closed space 6 and escapes from water outlet port 8. As the water passes through central cavity 2h, its moisture passes from the inner wall to the outer wall of central cavity 2h of hollow fibers 2 by molecular transfer, and humidification occurs by release of this moisture into the air. At the same time, adsorbed humidity passes through the walls of hollow fibers 2 and reaches the surface of the outer walls, and dries in contact with air. At this time because the ends of hollow fibers 2 are closely attached against closed spaces 5, 6, moist gases do not pass through the spaces between multiple aligned hollow fibers 2, and because the other ends [thereof] are closely attached against closed space 6, dry gases do not leak from the spaces between multiple aligned hollow fibers 2. Further, because the central parts of multiple aligned hollow fibers 2 are independent, the surface area [where] the outer walls of hollow fibers 2 contact the outer air I large, enabling the adsorbed and passed-through humidity to evaporate into the outer air with high efficiency.

0010

The following section describes a preferred embodiment of the present invention with reference to the drawings. Hollow fibers 2 are manufactured by means of the wet spinning method shown in Fig. 2. As shown in that drawing, the N-methyl-2-pyrrolidone (NMP) solution of aromatic polyether sulfone polymer used as raw material for the hollow fiber is placed in tank 20, and the copolymer suspension of tetrafluoroethylene and perfluorovinyl ether used as core liquid is placed in tank 21 and pressurized. Thus the raw material solution for spinning is sent through a pipe and ejected from the outer annular nozzle of concentric spinning nozzle 23, and the core liquid is ejected from the inside of spinning nozzle 23. As the ejected concentric fibers are guided by guides along congealing solution 24 formed of water, the solvents in the spinning raw material solution are removed from solution and the [material] congeals as fiber and is wound on a reel 25. When this fiber is cut to the required length, washed in water and dried, the finished hygroscopic hollow fiber 2 has central cavity 2h in the center of its cross sectional area as shown in Fig. 3.

0011

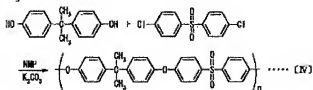
This hygroscopic hollow fiber 2 is incorporated into the gas humidifier shown in Fig. 1. The required number of hygroscopic hollow fibers 2 are arranged in parallel, and only the ends thereof are attached mutually with adhesive. One end is inserted into cap 3 having water intake port 7, and the other end is inserted into cap 4 having water outlet port 8, and they are tightly attached. Accordingly, a cross section of one end of hygroscopic hollow fiber 2 passes through closed space 5 to water inlet port 7, and a cross section of the other end passes through closed space 6 to water outlet port 8. The central cavities 2h of hollow fibers are unattached and are contained within mesh-like case 9 of rigid material. As a result case 9 is completely separated from closed space 5, and from closed space 6.

0012

(Synthesis of base material polymer for hollow fiber)

(1) Polymerization of aromatic polyether sulfone by means of the reaction shown in Chemical Formula IV below

[Chemical Formula 6]



0013

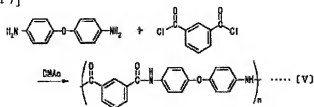
22.8g (0.10 mol) 2,2-bis (4-hydroxyphenyl) propane and 28.7g (0.10 mol) bis (4-chlorophenyl) sulfone were placed in a 1L flask to which was attached a cooler comprising a linked agitator, nitrogen conduit tube, and trap, and dissolved in 200 ml N-methyl-2-pyrrolidone. Then 18.0g (0.135 mol) anhydrous potassium carbonate and 100 ml toluene were added. The above procedures were performed while flowing nitrogen into the flask. The solution was heated to 140°-150°C under convective agitation for six

hours. Then it was reacted for four hours while removing the toluene from a trap, then raised to 190-195°C for one hour, and suction filtered while hot. After the filtrate was cooled, acetic acid was added until the solution color changed from dark green to light brown. This solution was strongly agitated and then slowly dripped into 4L methanol. This was filtered and dried to obtain aromatic polyether sulfone. Yield volume was 33.13g, yield rate 74.90%, and viscosity limit  $[\eta]$  0.747 dl/g (at 25°C in chloroform).

0014

(2) Polymerization of aromatic polyether amide (aramid) by means of the reaction shown in Formula V below

[Chemical Formula 7]



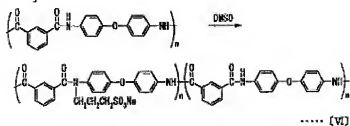
0015

A solution of diamino diphenyl ether 8.00g N-methyl-2-pyrrolidone 80ml was placed in a flask with attached nitrogen conduit tube and agitator and completely frozen by a dry ice-acetone bath in a nitrogen atmosphere, then isophthalate chloride 8.12 g added in solid form. Agitation was applied for 5 hours with the bath changed to an ice bath, then [the solution] was slowly poured into methanol 4000 ml, and the polymer was extracted. Then, the polymer was filtered, dried under low pressure, and an aramid was obtained. Viscosity limit  $[\eta]$  =1.67 (in dimethyl acetoamide dimethyl acetoamide, 30°C reference value 1.2 to 2.0 dl/g).

0016

(3) Alkyl sulfonation (-AS) of aramid by means of the reaction shown in Chemical Formula V below

[Chemical Formula 8]



0017

1.6g sodium hydride (60%) was added to 400 ml dimethyl sulfoxide (DMSO), and agitated for one hour at 75°C in nitrogen. The temperature of the system was then lowered to 40°C and 4.76g of the aforementioned aramid was thoroughly dried and added. This compound was agitated for 48 hours, then 4.89g 1-3-propane sultone was added in solid form and agitated for 24 hours at 40°C. The reactive mixture was placed in

tetrahydrofuran, and the resulting polymer was precipitated. The supernate was removed by decantation and the tetrahydrofuran was added to the residue and agitated, then filtered and dried to obtain aromatic polyetheramide alkylsulfone (alkyl sulfoneization 50-60%). The yield was 92.74%, FT-IR:3444 (amido), 1198 (S=O).

0018

(Spinning of hollow fiber)

Raw material solutions for spinning were prepared from the base polymers aromatic polyether sulfone (PES), aromatic polyether amide (aramid), and aromatic polyether amide alkyl sulfone (aramid-AS) according to the composition shown in Table 1, and core liquids were selected from among water, tetrafluoroethylene, and perfluorovinyl ether copolymer suspension solution (aqueous liquid manufactured by DuPont, trade name Nafion), and aqueous polysulfonated styrene-Na solution (PSSNa), as shown in Table 1.

0019

[Table 1]

Table 1.

|                        | Raw material solution for spinning |         |               | Core liquid                  |
|------------------------|------------------------------------|---------|---------------|------------------------------|
|                        | Polymer                            | Solvent | Concentration |                              |
| Preferred embodiment 1 | PES                                | NMP     | 15 wt%        |                              |
| Preferred embodiment 2 | PES                                | NMP     | 20            | Water                        |
| Preferred embodiment 3 | PES                                | NMP     | 20            | Nafion 5 wt % solution       |
| Preferred embodiment 4 | Aramid                             | NMP     | 15            | PSSNa 2 wt% aqueous solution |
| Preferred embodiment 5 | Aramid                             | NMP     | 15            | Water                        |
| Preferred embodiment 6 | Aramid = aramid (AS 12.5%)         | NMP     | 15            | PSSNa 2 wt% aqueous solution |
| Preferred embodiment 7 | Aramid = aramid (AS 30%)           | NMP     | 15            | PSSNa 2 wt% aqueous solution |

0020

By means of the wet spinning [method] shown in Fig. 2, raw material solutions for spinning according to each of the preferred embodiments were spun at feed rate 3.00 ml/min, core liquid feed rate 3.2 ml/min, and winding speed 7.12 m/min. These [results] were water washed and dried, to obtain hygroscopic hollow fibers according to preferred embodiments 1 through 7.

0021

(Manufacture of gas dryer)

Hollow fiber according to each preferred embodiment was cut to lengths of 15 cm, and hollow fiber bundles were made by gathering a number comprising a specified thickness into a parallel bundle, and setting only the ends with adhesive so that the resin did not enter the middle space. Both ends of [the fibers] were inserted into and bonded tightly with adhesive, to cap 3 having water intake port 7, and to cap 4 having water outlet port 8,

so that space opened between the end surfaces of hollow fibers 2 (see Fig. 1). Note also that case 9 previously formed of mesh etc. was inserted around the middle part of the bundle of hollow fiber between the two caps. Thus the body of the gas humidifier was completed.

0022

Water circulation path 11 is connected to water intake port 7 and water outlet port 8 which are part of the hollow fiber humidifier. Water tank 10 and water feed pump 12 are connected to water circulation path 11. Said tank 10 has water feed unit 13 through electromagnetic valve 19, in side of which are placed items such as flow switch 14 and electric heater 15. Said electric heater 15 and flow switch 14 are electrically connected to temperature and humidity controller 16. In addition, fan 17 flowing air towards hollow fiber 2 is placed near the side surfaces of hollow fiber 2.

0023

(Use of gas humidifier)

Water is passed through activated charcoal filter 18 into tank 10. Said activated filter 18 removes impurities such as chalk that are present in city water. Then, by means of water feed pump 12, water is circulated in the sequence water feed pump 12 → cap 5 → hollow fiber central cavity 2h → outlet cap 6 → tank 10. Meanwhile, fan 17 is sending away humidified air and moisture released from with hollow fiber 2 to the outer surface is dispersed in the air as moisture.

0024

Humidity is adjusted by changing the water temperature by temperature and humidity controller 16, changing the water feed pressure by water feed pump 12, or changing the air flow rate. These may also be used in combination. For example, the amount of humidification can be increased by increasing the water temperature, thereby increasing the partial pressure of water vapor. Similarly, changing the water feed pressure of water feed pump 12 can increase the partial pressure of water vapor in the water in hollow fiber central cavity 2h. And increasing the speed of rotation of fan 17, causes the pressure around hollow fiber 2 to drop, increasing the differential pressure between inside and outside and increasing the amount of humidification.

0025

(Gas humidifier humidification test)

Humid air was generated by directing interior air by means of fan 17 towards gas humidifier 1, prototyped from hygroscopic hollow fiber 2 according to [a] preferred embodiment. Intake gases having different temperature conditions were measured with respect to the differential pressure inside and outside of hollow fiber 2 and the permeation flow rate, or in other words the permeation flow volume. When the temperature of water inside the hollow fiber interior was increased the vapor pressure of water increases in proportion to the increase in water temperature. Because the partial pressure of water vapor on the outside of hollow fiber 2 is virtually constant, the vapor pressure differential increases. In proportion to this, the permeation flow flux increases, the humidification

volume also increases, and humidity increases. As a result the characteristics shown in Fig. 3 are obtained.

0026

### **Effect of the Invention**

As explained in detail above, a hollow fiber according to the present invention has a new function in that absorbs moisture as water passes through its interior, and releases it from its outer surfaces. For this reason, a gas humidifier according to the present invention that utilizes such hollow fibers is an extremely simple device and has extremely high-efficiency humidification capabilities that have not previously existed. Therefore this gas dryer can be used in a broad range of fields such as precision instrument industry, medicine, or semiconductor manufacturing equipment etc.

### **Simplified Explanation of Drawings**

Fig. 1 is a cutaway oblique view of a gas humidifier applying the present invention.

Fig. 2 is a drawing explaining the wet spinning for the purpose of manufacturing hygroscopic hollow fiber applying the present invention.

Fig. 3 is an oblique view of a hygroscopic hollow fiber applying the present invention.

Fig. 4 is a graph showing the relation between differential vapor pressure and permeation flow flux in permeable film.

### **Explanation of Symbols**

- 1... high polymer film humidifier
- 2... hollow fiber
- 2h... central cavity
- 5, 6... closed space
- 7... water intake port
- 8... water outlet port,
- 9... case
- 11... water circulation path
- 12... water feed pump
- 13... water feed unit
- 14... flow switch
- 19... electromagnetic valve
- 20,21.. tank
- 23... spinning nozzle
- 24... congealing solution
- 25... reel

Fig. 3

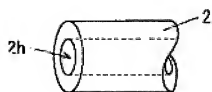


Fig. 1

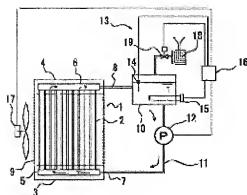


Fig. 4

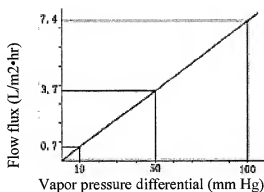
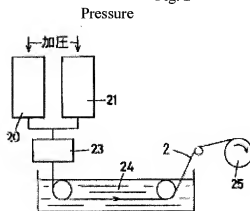


Fig. 2



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## F Terms (Reference)

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NA17 PA10 PB06 PB17 PB65  
PB70 PC01 PC41 PC80  
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